

As the panelists learn to recognize gas phase odor, flavor improvements effected by these additives can be measured.

Efforts are being continued to reduce the drying effect of efficient, especially paper, filters. It may be that liquids like triacetin and propylene glycol prevent excessive moisture adsorption by filling some of the paper pores.

Minutes of

SUPERVISORS' MEETING

August 29, 1958

Present: Dr. R. C. Sproull  
Dr. C. V. Mace, Jr.\*  
Dr. R. B. Seligman  
Mr. J. D. Hind (Chairman)  
Mr. C. C. Cosby, Jr.\*  
Mr. H. J. Dooley\*  
Mr. G. H. Sharp, Jr.  
Mr. J. C. Holmes  
Mr. S. W. Pleasants  
Mr. P. L. Gager, Jr.  
Mr. J. T. Butler  
Mr. F. E. Resnik  
Mr. W. H. Danker  
Mr. A. B. Clarke  
Mr. D. G. Michels (Secretary)

ADMINISTRATIVE

Mr. Cosby reminded all supervisors that they should examine the time cards of the members of their groups to make certain the correct account numbers are used. A great deal of the delay in typing can be traced to poor penmanship. The supervisors were asked to urge the members of their groups to write more legibly.

Dr. Seligman announced that five papers have been approved for the Tobacco Chemists' Conference. The authors have been notified.

Dr. Sproull announced that all authors should submit a written list of their contributions to any paper so that authorship may be established.

Dr. Mace announced that Mr. Budne has been retained for one day per week for the rest of the year. Anyone with problems he would like to discuss with Mr. Budne should contact Dr. Mace. Further, Dr. Mace would like to have a list of subjects that Mr. Budne could discuss in group meetings with our technical people.

SUMMARY

The gas phase smoke of Tareyton cigarettes, with and without the filter, was gas chromatographed at room temperature. Reductions in peak areas of up to 45% were observed in the chromatogram of the filtered smoke. Two small chromatographic peaks from the non-filtered smoke were not detected in the filtered smoke chromatogram.

\* Administrative only.

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Philip Morris cigarettes treated with 4% glycerine and 4% TEG were compared by 40 laboratory smokers for mildness, sweetness, and preference. Of those smokers stating a preference, the 4% TEG cigarette was significantly preferred. The 4% TEG cigarette was found to be milder and sweeter than the 4% glycerine cigarette at about the 10% level.

-2-

Paper chromatographic data indicate some decomposition of alkaloids takes place under the conditions of high temperature gas chromatography.

Recent results show less formaldehyde in the smoke of king-sized PM cigarettes cased with TEG than was found earlier in the smoke of regular PM's cased with glycerol.

Gas chromatographic patterns were qualitatively similar for fractions obtained from acidified aged and unaged tobacco by sweeping with nitrogen at elevated temperatures.

A DNP derivative, thought to be that of a C<sub>8</sub> or C<sub>9</sub> carbonyl compound, was observed on a paper chromatogram of the steam volatile neutral fraction from the petroleum ether extract of aged tobacco.

Qualitative analysis shows that glycerol is the principal humectant in the new Tareyton cigarette. Some propylene glycol is also present. A white inorganic material, not found in previous tobacco leaf or filler extracts, was observed in the aqueous extract of the Tareyton filler.

Both glycerol and propylene glycol were detected in the extracts of the plugs of competitive mentholated cigarettes (Salem, Newport, Oasis, Kool). A peak for propylene glycol was not observed on the gas chromatogram of an extract of Mayfield plugs.

The size distribution of smoke from several different kinds of cigarettes, a cigar, and a pipe were determined by the settling technique.

Dilution has a profound effect on the mass mean diameter of cigarette smoke.

It has been calculated that the number concentration of cigarette smoke at the end of the cigarette is about  $1 \times 10^{10}$  particles per milliliter. At this concentration there would be a decrease to one-tenth this concentration in 0.03 second.

Reproducible results were obtained in the analysis of gas phase smoke from PM cigarettes when the weight range was the only variable held constant.

The present IEF program is being expanded to produce analyses in mole per cent of the constituents present in the computed spectra.

Analysis of the particulate phase of the smoke from cigarettes containing 49 milligrams of TEG per cigarette showed that an average of 6.2 mg. of TEG per cigarette, or about 12%, is being transferred to the smoke stream.

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169 Sugar (reducing): The samples analyzed were priority bright leaf, special test cigarettes, and known sugar samples.

03. FILLER

0301 Determination of Humectant in Filler (Martin, Carpenter)

Schweitzer regular reconstituted tobacco contains 2.8% glycerol.

-3-

No significant differences were noted in the CO, CH<sub>4</sub>, ethene, and acetylene contents of the gas phase smoke from cigarettes containing different humectants (glycerine, TEO, propylene glycol, and water).

No significant differences were noted in the CO, CH<sub>4</sub>, ethene, acetylene, and isoprene contents of the gas phase smoke from Marlboro cigarettes, with or without filters.

Isopropyl alcohol is a satisfactory solvent for a polarographic system which is being developed for the qualitative and quantitative analysis of polyphenols. Ethyl alcohol is not a satisfactory solvent for this system.

More reproducible results and easier oxygen removal are achieved in the analysis of quercetin when a 50/50 water-isopropyl alcohol mixture is used instead of 100% isopropanol.

The gas phase smoke from carbon filtered cigarettes contained 53% less CO, 44% less methyl chloride, 54% less isoprene, 51% less benzene, and 62% less toluene than the gas phase smoke from non-filtered cigarettes. The non-filtered smoke contained 16% less water than the filtered smoke.

The triacetin content of Tareyton plugs (carbon portion) is 2% by weight. The triacetin content of Winston plugs is about 7%.

The ether extract of glass wool contained a silicone.

Infrared analysis of esters obtained from Mr. Hind and fractionated by gas chromatography showed the presence of diethyl succinate, triethyl citrate, possibly diethyl malonate, and possibly a keto or thio ester.

An adhesive sample from Polymer, Inc., contained only CaCO<sub>3</sub>, while a competitor's adhesive sample being tested contained CaCO<sub>3</sub> and silica. The Polymer sample contained more butyl rubber than did the competitor's sample.

In a paired comparison test, no significant difference in sweetness, mildness, and preference was found between PM cigarettes treated with 4% glycerine and PM's treated with 4% propylene glycol. More judges found the 4% glycerine cigarettes sweeter in flavor.

In a triangle test, no significant difference was found between AT&T cigarettes and BL cigarettes at the 30% level (70% PM filler).

More evidence is being accumulated that triacetin improves smoke flavor by increasing its mildness.

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Previously we reported that DL containing approximately 28, 14, and 7%  $\text{CaCO}_3$  delivered less tars than did regular DL. A subsequent sample containing 20%  $\text{CaCO}_3$  was checked for tar delivery. This sample showed no difference from regular DL. Additional smoking machine runs are being made to check this situation.

-4-

So-Lonita cigarettes with triacetin treated filters were found to be more pleasant and less strong and irritating than So-Lonita cigarettes without triacetin treated filters.

Panel judges were able to differentiate among three levels of triacetin, 20%, 13.4%, and 6.7%; the higher levels were the most pleasant.

O-So-Lonita cigarettes with added filter flavors received more positive responses in a preliminary handout test than did O-So-Lonita cigarettes without added filter flavors. The filter cigarettes flavored with a composite of coumarin, maple, anise, and rum received the most positive responses.

In a paired comparison test, 13 of 24 judges preferred So-Lonita to Kent.

In a triangle test, no difference could be detected between Marlboros stored for nine weeks in air and Marlboros stored for nine weeks in nitrogen.

No flavors were absorbed by PM filler from flavors painted on the stamps of PM packs.

Eight flavors have been found which mask the isoprene odor.

Odor and color changes occur when optimum proportions of diethanolamine and triethanolamine are blended with acetaldehyde in vitro. Plugs impregnated with DEA and TEA turned orange when Millipore-filtered gas phase smoke was drawn through them.

The nicotine and tar contents of the smoke of burley tobacco show significant positive correlation with each other and with the content of total alkaloid, total volatile bases, total nitrogen, and petroleum ether extract. Significant negative correlations were observed between the smoke constituents and the total ash content of the leaf.

The correlation coefficients for several chemical constituents of flue-cured tobacco, calculated by the USDA, were recalculated on an alcohol extract-free basis. These coefficients indicate a negative correlation between total ash and the nitrogenous constituents. This gives additional evidence that the large variations in the soluble constituents (e.g., sugars) mask associations among the constituents having smaller variations.

The reducing sugar content of O-67 tobacco was decreased approximately 25% and the TVB content was decreased approximately 20% during forced aging in nail kegs. Significant smaller reductions were observed in the petroleum ether extract, total alkaloid, total ash, and total nitrogen contents.

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1) A filter which contains four discs - staple cellulose acetate, cotton, glass fibers, and staple cellulose acetate. The discs are placed in the filter so that the fibers are perpendicular to the stream of smoke.

2) Staple acetate chopped in the Waring blender and cut as a disc.

-5-

The alkaloid content of loosely packed flue-cured tobacco was reduced approximately 25% by treatment with ammonia gas and superheated steam in the simulated Legg apparatus.

The alkaloid content of a sample of flue-cured tobacco was reduced approximately 10% by manual removal of the heavier leaf.

Six grades of Venezuelan burley tobacco, 1957 crop, are characterized by large alkaloid, nitrogen, and TVB contents. They would be expected to produce a strong and irritating smoke.

The best synthetic smoking material so far produced is a mixture of equal parts of tobacco stem pulp and condenser paper flavored with PM flavors and tobacco extract.

The lowest tar delivery yet obtained (7.0 mg., 62% efficiency) was from an O-So-Lonita plug on a special filter, ventilated cigarette. The next lowest (7.7 mg., 50% efficiency) was from a paper-rayon blend filter.

A carbon wool filter reduced the gas phase of smoke appreciably.

The refining time of soda ash-cooked, solubles-retained pulp was equal to the refining time of current production pulp; the refining time of the latter was less than the refining time of previously tested production pulp.

Initial tests on binder made from unwashed pulp show that viscosity is reduced in a day or two by degradation but that there is little effect on the physical characteristics of the cast film.

BL made with binder from burley stem pulp has been reported to have a 30% higher burning rate than specifications call for. Recent Pilot Plant controls have a slightly higher burning rate than the burley. The apparent high burning rate of the burley binder BL is probably insignificant.

Tensile and elongation measurements on Pilot Plant BL made with technical and purified glyoxal have not demonstrated that glyoxal gives a stronger or more elastic product.

The tensile strength of production BL did not change during storage periods of up to eighteen months. There were no significant changes in equilibrium moisture content during such storage periods.

Microscopic examination of cotton linters which were suspended and beaten in 200 ml. of a mixture of water, acetic acid, and sulfuric acid showed the fibers had fibrils projecting from them.

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fibrous structures produced from the growth of fungi within filters and fibers from grape leaves.

Evaluation of Benson & Hedges cigarettes containing Estron and cotton filters (Miller, Maxwell, Danker)

In a paired comparison test by 40 judges, there was no significant difference found in preference between Benson & Hedges

Dual filters of paper-cotton, paper-Parliament, and cotton-Parliament did not show any advantage over the expected filtration efficiency from the sum of the individual filters.

The Newport filter has a lower denier filament than before; its filament now has about the same denier as the Old Gold filament.

Kent 70 now has a dogbone-shaped filament.

The Mayfield recessed and flush filters contain "X"-shape filaments and rice starch additive.

Liggett & Myers' cigarette filters continue to have a duller yarn than do the other cigarette filters.

The separation of a crude ester fraction from salt-saturated liquors can be accomplished reasonably well in a small basket type centrifugal.

Schweitzer low nicotine BL has approximately 50% less nicotine than either Schweitzer regular BL or Philip Morris product BL.

The average recovery of R.K.S. mixed with Marlboro filler at 1%, 3%, 5%, and 12% levels was 84%.

The amount of R.K.S. present in ten Marlboros, randomly selected from our current production, was much less than was expected. The particle size of R.K.S. in the cigarettes was much smaller than the particle size of R.K.S. sampled at the blending line.

A check of the puff volume for the eight port constant volume type smoking machine showed an average variance ( $S^2$ ) of only 0.07 cc/puff for the eight ports. The average puff volume, with all resistances except that of the cigarette measured, was 35.9 cc/puff. The average puff volume, when a piece of glass capillary tubing was used to simulate a cigarette, was 35.4 cc/puff.

A study was begun to determine the moisture content of the "total solids" as collected on filter discs by the P.T.C. procedure and to determine the amount of material lost by desiccation. Preliminary results showed an average moisture content of 10.7% and a loss on desiccation of 18.7%.

While countercurrent extraction is theoretically better than single partitioning, for the separation of nicotine from the "total solids" collected on glass filter discs, a single partitioning is sufficient.

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Cigarettes have been prepared which contain 30% fruit fiber and 70% Marlboro filler. The Marlboro filter is attached. These cigarettes are being evaluated to determine smoking characteristics of the fruit fiber.

4003 Process for a Low Tar Cigarette (Michels, Long)

A cigarette prepared as follows delivered 6.5 mg. tar/cigt.:

-7-

TECHNICAL

21-0101 Chemical Composition of the Gas Phase of Smoke (Murrill)

The gas phase smoke of Tareyton cigarettes, with and without the filter, was gas chromatographed at room temperature. Reductions in peak areas of up to forty-five per cent were observed in the chromatogram of the filtered smoke. Two small chromatographic peaks from the non-filtered smoke were not detected in the filtered smoke chromatogram. Duplicate experiments showed similar results. The smoke of single cigarettes was used.

Discussion: The data qualitatively check the data given in C & E News. Quantitative examination will be the next step. Mass spectrometric data confirm the C & E News data quantitatively with respect to isoprene.

Some high filtration cigarettes have an odor of isoprene. This compound is partially removed by activated carbon. Since isoprene has a low threshold value, it would be expedient to remove as much of it as possible in a low tar cigarette.

21-0102 Chemical Composition of the Particulate Phase of Smoke (Poindexter)

Additional paper chromatographic data indicate some decomposition of alkaloids takes place under the conditions of high temperature gas chromatography.

21-0104 Chemical Composition of Whole Smoke - Formaldehyde (Martin, Carpenter)

The effects of different humectants on the formaldehyde content of cigarette smoke are being determined. TEG-cased PM king size cigarettes show a lower formaldehyde level in the smoke than that obtained earlier from glycerine-cased PM regular cigarettes (32 micrograms compared to 45 micrograms per cigarette).

21-0201 Qualitative and Quantitative Analyses of Tobacco Leaf Compounds (Brunot, Edmonds, Greene, Guvernator, Carpenter)

Acidified tobacco samples are being swept with nitrogen in heated columns. The effluent vapors are being trapped. One fraction from an aged sample had a characteristic aged tobacco odor. Gas chromatographic patterns obtained for fractions from aged and unaged tobacco were qualitatively similar. Parallel experiments are being carried out in an effort to compare the volatile constituents quantitatively.

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SUPERVISORS MINUTES

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The steam volatile neutral fraction from the petroleum ether extract of aged tobacco was examined by gas chromatography before and after reaction with the dinitrophenylhydrazine reagent for carbonyls. A derivative, thought to be that of a C<sub>8</sub> or C<sub>9</sub> carbonyl compound, was observed on a paper chromatogram, although no qualitative differences were noted on the gas chromatograms.

On the basis of chromatographic, chromogenic, and spectral data, attempts are being made to develop quantitative methods of analysis for the principal polyphenolic compounds in tobacco. Chromatographic, polarographic, and spectrophotometric techniques are being used in cooperation with the Instrument Section.

21-0301 Determination of Humectants on Filler (Carpenter, Martin)

Qualitative analysis shows that glycerol is the principal humectant in the new Tareyton cigarette. Some propylene glycol is also present. A white inorganic material, more soluble in cold water than hot, was observed in the aqueous extract of the filler. This has not been found in previous tobacco leaf or filler extracts. The substance will be investigated further.

21-6901 Miscellaneous Small Projects from Within the Division (Carpenter, Martin, Poindexter)

Extracts of the plugs of competitive mentholated cigarettes (Salem, Newport, Oasis, Kool) were examined by paper chromatography. The presence of both glycerol and propylene glycol was detected. The propylene glycol explains a large peak on the gas chromatograms of similar extracts. This peak was not observed on the gas chromatogram of an extract of Mayfield plugs.

22-0105-00 Physical Mechanism of Smoke Formation (Hardcastle, Holmes)

The size distribution of smoke from several different kinds of cigarettes, a cigar, and a pipe have been determined by the settling technique. The results have been obtained from only one run on each cigarette, and therefore the limits of variation have not yet been established.

<u>TEST CIGARETTE</u>	<u>MASS MEAN DIAMETER</u>
Parliament with spun acetate filter	1.05 micron
Bright tobacco (filtered)	1.05 "
Turkish tobacco (filtered)	0.80 "
Burley (unfiltered)	1.02 "
Tareyton (filtered)	0.98 "
Hit Parade	0.96 "

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<u>TEST CIGARETTE</u>	<u>MASS MEAN DIAMETER</u>
Cigar	1.01 micron
Pipe Smoke	0.86 "

The smoke in all of the above tests was measured without dilution.

The effect of dilution on cigarette smoke has been measured. Smoke from regular Philip Morris cigarettes was diluted as quickly as possible after issuing from the cigarette, then sampled with the cascade impactor. The data obtained from this experiment are as follows:

<u>DILUTION RATIO</u>	<u>MASS MEAN DIAMETER</u>
300:1	0.2 micron
45:1	0.5 "
30:1	0.6 "
22 1/2:1	0.64 "
undiluted	0.92 "

It can be seen that dilution has a profound effect on the mass mean diameter. This is in agreement with the prediction based on Smoluchowski's equation that agglomeration takes place at a high rate when the number concentration of particles is high. Based on the amount of material collected and the size of particles, we have calculated that the number concentration of cigarette smoke at the end of the cigarette is about  $1 \times 10^6$  particles per milliliter. At this concentration there would be a decrease to one-tenth this concentration in 0.03 second.

23-0101-03 Development of Methods for Determining Quantitatively the Gas Phase Constituents of Smoke (Varsel, Bell, Dodson)

Analysis of gas phase smoke from Philip Morris cigarettes showed reproducible results when the weight range was the only variable held constant. That is, variables such as soft spots and resistance to draw were not measured.

Work has been resumed on the removal of  $\text{CO}_2$  and  $\text{H}_2\text{O}$  from gas phase smoke. Success in this removal would make possible the analysis of a larger number of the smoke constituents, particularly after computation on the IBM 650.

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The present IBM program is being expanded to produce analyses in mole per cent of the constituents present in the computed spectra.

23-6101-00 Instrument Maintenance. Research Division (McMrell, G. Smith)

The mass spectrometer has been repaired and is back in full operation.

The usual routine service was performed during this period. In addition, a Brown recorder and integrator were assembled in a chassis as one unit and placed on casters for mobility. The integrator and recorder are presently being used by the Gas Chromatography Group.

23-6202-00 Instrumentation. Research with the Infrared Spectrophotometer (Bill, Vilcins)

Cigarettes containing 49 mg. of TEG per cigarette were smoked, and the particulate phase was analyzed for TEG content. Analyses showed that an average of 6.2 mg. of TEG per cigarette, or about 12%, is being transferred to the smoke stream.

The gas phase smoke from cigarettes containing different humectants (glycerine, TEG, propylene glycol, and water) was analyzed with the infrared spectrophotometer. No significant differences were noted in the CO, CH<sub>4</sub>, ethane, and acetylene contents of these various cigarettes.

No significant differences were noted in the gas phase smoke (CO, CH<sub>4</sub>, ethene, acetylene, and isoprene) from Marlboro cigarettes, with or without filters.

23-6203-00 Instrumentation. Instrumental Research with the Ultraviolet Spectrophotometer (Kuhn)

Work on the colorimetric determination of "NO<sub>2</sub>" in cigarette smoke continued. An additional trap in the collection "train" showed that all of the "NO<sub>2</sub>" was being collected in the 2-liter flask.

23-6204-00 Instrumentation. Research with Instruments. Polarograph (Kuhn)

A polarographic system is being developed for the qualitative and quantitative analysis of polyphenols. Ethanol is not a satisfactory solvent for this system because it is air oxidized to acetaldehyde, which gives a half-wave in the same region as the polyphenols. Isopropyl alcohol, on the other hand, is oxidized to acetone, which gives a half-wave far removed from the polyphenols. Isopropyl alcohol, therefore, is a satisfactory solvent

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Analysis of quercetin showed that more reproducible results and easier oxygen removal are achieved when a 50/50 water-isopropyl alcohol mixture is used instead of 100% isopropanol. Attempts will be made to analyze polyphenols eluted from paper chromatograms.

23-6901-00 Miscellaneous Small Projects within the Division  
(Bill, Varsel, Dodson, Bell, Kuhn, Vilcins)

Cigarettes with activated carbon filters: Cigarettes with carbon filters obtained from the Development Division were smoked, and the gas phase smoke was analyzed mass spectrometrically. Cigarettes without filters were carried through the same procedure. Results showed that the gas phase smoke from the carbon filtered cigarettes contained 53% less CO<sub>2</sub>, 44% less methyl chloride, 54% less isoprene, 51% less benzene, and 62% less toluene than the non-filtered smoke. The non-filtered smoke contained 16% less water than the filtered smoke. These are averages of three analyses of each cigarette type. The experiments will be repeated.

Triacetin on Tareyton and Winston plugs: The triacetin content of Tareyton plugs (carbon portion) was found to be 2% by weight.

The triacetin content of the Winston plugs is about 7%.

Glycerine on filter plugs: Analysis of the extracts from the filter plugs from Salem, Newport, Oasis, and Kool all showed the presence of glycerine, both by infrared and paper chromatography analysis. It is quite possible that the humectant is diffusing from the filler to the plugs.

23-6903-00 Miscellaneous Services (Bill, Kuhn, Vilcins)

Silicone on glass wool: Analysis of the ether extract of glass wool showed the presence of a silicone.

Esters fractionated by gas chromatography: Esters (from Mr. Hind) were fractionated by the Gas Chromatography Group and analyzed with the infrared spectrophotometer. Results show the presence of diethyl succinate, triethyl citrate, possibly diethyl malonate, and possibly a keto or thio ester. It was suggested that these be analyzed with the mass spectrometer if further information is desired.

Alkaloids in Tobacco: Fifty samples were analyzed for individual alkaloids during this period. The difficulty with the "blank" appears to be solved since no absorbance peak was obtained when the Whatman #1 paper was treated in a manner analogous to the preparation of an alkaloid.

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23-9703-00 Service to Polymer, Inc. (Bill, Kuhn, Vilcins)

Infrared analysis showed that the adhesive from Polymer was different from the competitor's adhesive in filler content. The Polymer sample contained only  $\text{CaCO}_3$ , while the competitor's sample contained  $\text{CaCO}_3$  and silica. More butyl rubber was found in the Polymer sample than in the competitor's sample.

23-9803-00 Service to Milprint, Inc. (Morrill, O. Smith)

A prototype gas diffusion apparatus has been constructed for Milprint, Inc. It employs thermistors and has been calibrated to measure  $\text{CO}_2$  in  $\text{O}_2$  and  $\text{O}_2$  in  $\text{CO}_2$ .

24-0202 Humectant Comparison (Miller, Maxwell, Danker)

In a paired comparison test, forty-three judges found an insignificant difference in sweetness, mildness, and preference between Philip Morris cigarettes treated with 4% glycerine and PM's treated with 4% propylene glycol. More judges found the 4% glycerine cigarettes sweeter in flavor. The mouth emollient effect of propylene glycol plus regular humectant will be further investigated with factory-made cigarettes.

24-1302 Improvement in formulation of BL (Miller, Maxwell, Danker)

In a triangle test, a panel of our twelve best judges found an insignificant difference between ATBL cigarettes and BL cigarettes at the 30% level (70% Philip Morris filler). Several new flavor improvements were recommended at a conference with the Development Group. An accelerated program was planned.

24-2101 Development of New Filter Materials (Maxwell, Danker, Miller)

Evaluation of triacetin in filters: More evidence is being accumulated that triacetin improves smoke flavor by increasing its mildness.

Twenty judges were able to distinguish between So-Lonita cigarettes and So-Lonita cigarettes that had extra triacetin added to the filter. The So-Lonita cigarettes without added triacetin were found to be stronger and more irritating. The So-Lonita cigarettes with triacetin treated filters were found to be more pleasant. Results show that our judges were able to differentiate among three levels of triacetin, 20%, 13.4%, and 6.7%; the higher levels were the most pleasant.

Evaluation of flavor additives in extra low tar cigarettes: O-So-Lonita cigarettes with and without added filter flavors were compared. In a preliminary handout test, the flavored filter

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cigarettes received more positive responses. The filter cigarettes flavored with a composite of coumarin, maple, anise, and rum received the most positive responses.

24-8101 Service to Brand Comparison Studies and Intelligence  
(Miller, Maxwell, Danker)

Kent vs So-Lonita: In a paired comparison test, thirteen of twenty-four judges preferred So-Lonita to Kent.

Kent vs So-Lonita vs Hit Parade: A profile test is being run on the three cigarettes for bitterness, sweetness, fruitiness, flavor, pleasantness, unpleasantness, hotness, dryness, tongue bite, throat irritation, and after taste.

Discussion: Dr. Seligman suggested that the cigarettes be smoked without filters before they are smoked with filters in order to compare the tobacco flavors.

24-8204 Service to Operations Department (Miller, Maxwell, Danker)

Marlboro cigarettes stored in air vs Marlboro cigarettes stored in nitrogen: In a triangle test, twelve of our best smoking judges could not detect a difference between Marlboro cigarettes stored for nine weeks in air and Marlboros stored for nine weeks in nitrogen.

Evaluation of Philip Morris pack aroma - Flavors painted on the stamp only:

Odor panel evaluations were made of Philip Morris packs on which the stamps were painted with several flavor formulas. After three weeks of aging under various conditions, the same two formulas were preferred by our panel and by Mr. Hatcher. Since some of the flavors were too weak, higher flavor levels were prepared and are being evaluated. No flavors were absorbed by the filler.

Masking gas phase flavor: We have determined the threshold and a moderate level of acetone and furan for the purpose of masking these unpleasant flavors in smoke.

We have found eight flavors which can mask the isoprene odor. These are sage, hay, spruce, nutmeg, cade, orris root, currant, and apple. First tests show that judges note a more pleasant aroma in the Millipore filtered smoke. About 20 other flavors tested allow the isoprene to show through even at moderately high flavor levels.

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Ethanolamines vs Acetaldehyde: At the suggestion of Mr. Loyal Davis we are studying the neutralizing effect of diethanolamine and triethanolamine on acetaldehyde. Dramatic odor and color changes were found when optimum proportions were blended in vitro. Plugs impregnated with TEA and TEA turned orange when Millipore-filtered gas phase smoke was drawn through them. First tests showed taste improvement.

Mayfield Advertising Aids: A conference was held with our Mayfield advertising agency. We were asked to add to Mr. Hatcher's suggestions for Mayfield characteristics which may be exploited. We shall attempt to add more suggestions based on descriptive panel evaluations. The mouth watering and moisturizing effects will be given special study.

25-0203-00 Correlation of Chemical Composition, Physical Properties, and Smoke Analysis with Leaf Quality  
(Bass, Sharp, Mason)

The nicotine and tar contents of the smoke of burley tobacco show significant positive correlation with each other and with the content of each of the following leaf constituents: 1.) total alkaloid, 2.) total volatile bases, 3.) total nitrogen, and 4.) petroleum ether extract. Significant negative correlations were observed between the smoke constituents and the total ash content of the leaf.

The correlation coefficients for several chemical constituents of flue-cured tobacco, calculated by the USDA, have been recalculated on an alcohol extract-free basis. These coefficients indicate a negative correlation between total ash and the nitrogenous constituents. This gives additional evidence that the large variations in the soluble constituents (e.g., sugars) mask associations among the constituents having smaller variations.

25-0204-00 Development of Improved Leaf (Bass, Mason)

Reports have been prepared concerning the inspection tours of experimental farms in the flue-cured and burley producing areas.

25-0702-00 Procedures and Conditions for Accelerated Aging  
(Crayton, Mason)

The reducing sugar content of O-67 tobacco was decreased approximately 25% and the TVB content was decreased approximately 20% during forced aging in nail kags. Significant smaller reductions were observed in the petroleum ether extract, total alkaloid, total ash, and total nitrogen contents. Smoke analyses of the forced-aged and unaged tobaccos are in progress.

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25-8204-00 Services to Operations Department (Crayton, Mason)

The alkaloid content of loosely packed flue-cured tobacco was reduced approximately 25% by treatment with ammonia gas and superheated steam in the simulated Legg apparatus.

Additional work is planned with tobacco packed at hogshead density.

25-8401-00 Services to Leaf Departments: (Bass, Sharp, Mason)

The alkaloid content of a sample of flue-cured tobacco was reduced approximately 10% by manual removal of the heavier leaf.

The chemical leaf analysis of four grades of flue-cured tobacco from the Dominion Leaf Tobacco Co. helped the Leaf Department decide to purchase large amounts of three of the grades.

A large number of stemmery samples and market samples from Georgia, Florida, and South Carolina have been studied. Most of this tobacco is of good quality and should have mild smoking characteristics.

Six grades of Venezuelan burley tobacco, 1957 crop, were studied. These tobaccos are characterized by large alkaloid, nitrogen, and TVB contents. They would be expected to produce a strong and irritating smoke.

31-4002 Development of an Entirely New Cigarette (Westbrook, Clarke)

A preliminary study has been made of several types of paper (kraft, condenser, and highly purified wood fibers) to determine whether a good synthetic smoking material could be produced from them. The best smoking material so far produced is a mixture of equal parts of tobacco stem pulp and condenser paper flavored with PM flavors and tobacco extract.

31-4002 New Cigarette Development (Toepffer, Wright, Michels, Long)

Spun acetate filter: Nothing new to report.

31-4003 Low Tar Cigarette (Wright, Michels, Long)

Six lots of hand-combined cigarettes have been made with a tar delivery below 10 mg. per cigarette. The lowest delivery (7.0 mg., 62% efficiency) was with an O-So-Lonita plug on a special filler, ventilated cigarette. The next lowest (7.7 mg., 50% efficiency) was with a paper-rayon blend filter.

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A carbon wool filter reduced the gas phase of smoke appreciably.

We are still trying to improve the burning characteristics of synthetic filler without increasing the carbon content.

Two lots of low tar cigarettes were furnished the Flavor Group for flavor studies.

33-1002      Development of Sheet Material from Stems and Stalk  
(Westermann, Clarke)

Twenty cigarettes with the paper made from bleached tobacco stalk have been prepared. These cigarettes will be smoked to determine if the stalk paper has objectionable taste or irritation.

34-1301      Improvement in BL as Manufactured (Scherr, Long)

The refining time of soda ash-cooked, solubles-retained pulp was equal to the refining time of current production pulp; the refining time of the latter was less than the refining time of previously tested production pulp.

Initial tests on binder made from unwashed pulp show that viscosity is reduced in a day or two by degradation but that there is little effect on the physical characteristics of the cast film. The effect of aging on the binders is being investigated along with a study of means of improving the situation.

It has been reported that BL made with binder from burley stem pulp has a 30% higher burning rate than specifications call for. Recent Pilot Plant controls have a slightly higher burning rate than the burley. The apparent high burning rate of the burley binder BL is probably insignificant.

34-1301      Improvements in BL (Hind)

Tensile and elongation measurements on Pilot Plant BL made with technical and purified glyoxal have not demonstrated that glyoxal gives a stronger or more elastic product. Measurements were made at the 20-24% moisture level and at the 12-15% moisture level on test specimens 6" and 18" long.

Tensile data obtained on samples of production BL stored for up to 18 months were compared with data obtained within a few days of the production date. The tensile strength of the BL has not changed in periods ranging up to 18 months, and there have been no significant changes in equilibrium moisture content.

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34-2101 Development of a New Filter Material (Westbrook, Clarke)

An investigation is underway to determine whether cellulose fibers can be fibrillated chemically in a mixture of water, acetic acid, and sulfuric acid. Five grams of cotton linters were suspended and beaten in 200 mls. of this ternary mixture. Microscopic examination of the beaten material showed the fibers had fibrils projecting from them.

34-2101 Development of an Improved Filter Material (Super)

At the last supervisors' meeting it was reported that dual and triple filters in which one of the elements was a paper filter showed better filtration efficiencies than would be expected from the sum of the efficiencies of the individual elements.

Further results, concerning dual filters of paper-cotton, paper-Parliament, and cotton-Parliament, did not show any advantage for the dual filters over the expected filtration efficiency from the sum of the individual filters.

More dual filters have been prepared so that we can draw more definite conclusions as to their value and can determine whether the position of the elements with respect to each other may affect the filtration.

35-0501 Development and Application of Microscopic Techniques (Johnson, Clarke)

Twenty-seven cigarette filters (August pick-up), our own and competitive brands, were examined for Comparison of Brands studies and for Intelligence. The following facts were noted:

1. Newport has a lower denier filament, about the same denier as the Old Gold filament.
2. Kent 70 now has a dogbone-shaped filament.
3. The Mayfield recessed and flush filters contain "X"-shape filaments and rice starch additive.
4. Liggett & Myers' cigarette filters continue to have a duller yarn than do the other cigarette filters.

Several suitable cigarette filter materials (dentist napkin, metalized fiber glass fabric, and web-rolls) have been examined for material content.

35-1304 ATBL (Westermann, Clarke)

A smoking test was run by the Flavor Section on two sets of cigarettes. One set contained 30% unflavored ATBL and 70% PM filler; the other contained 30% BL and 70% PM filler. None of

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the 12 persons on the panel could distinguish between the two cigarettes.

Work is continuing on the development of a flavor within the stem pulp by oxidation of the lignin and/or esterification of the organic acids with iso-aryl alcohol. Likewise, work continues on the addition of flavors to ATBL, as recommended by the Flavor Section.

35-2301-00 Flavors from Tobacco Waste (Burnett)

The separation of a crude ester fraction from salt-saturated liquors can be accomplished reasonably well in a small basket type centrifugal. Some alterations in the process steps leading up to this separation may be necessary in order to eliminate impurities which, in some cases, make the crude ester too heavy and viscous for separation.

71-0103 Quantitative Composition of the Particulate Phase of Smoke (Wickham, Stewart, Pleasants)

The following number of smoke analyses were completed during the period: 528 tar; 58 nicotine.

The evaluation of the automatic charline detector is being continued. For ten tests the variance of the manual cutoff was lower than that of the automatic charline detector; however, more data must be obtained before we can be sure this difference is statistically significant.

Griffith steam distillation of nicotine in smoke: Ninety-seven smoke solution samples were distilled and analyzed spectrophotometrically during this period.

71-0204 and 71-0304 Leaf and Filler Analyses (Nedlock, Wiley, Pleasants)

247 Sugar: Priorities, Intelligence Test, Special cigarettes, BL.

85 Ash: Priorities, BL

162 Petroleum Ether: Priorities, Aging Samples, BL.

138 Total Nitrogen: Priorities, Stemmary.

115 T.V.B.: Priorities

3 Triacetin: Intelligence Test, Special.

48 Menthol: Intelligence Test, Special cigarettes.

60 BL: Intelligence Test, Special.

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352 Nicotine: Product Audit, Intelligence Test, Specials,  
BL, Bright.

1210 Total samples

71-1301 Physical Testing of BL Products (Wickham, Stewart,  
Pleasants)

The chemical analyses of Schweitzer regular BL, Schweitzer low nicotine BL, and Philip Morris production BL show that Schweitzer low nicotine BL has approximately 50% less nicotine than either Schweitzer regular BL or Philip Morris production BL.

71-6903 Miscellaneous Laboratory Tests (Westbrook, Nedlock,  
Pleasants)

Paper Chromatography (Individual Alkaloids): Every effort is being made to increase the speed and precision of this analysis. The equipment has been moved away from a live steam line to a different work bench. The glassware is being etched numerically to make it easier to keep track of the samples. The reagents are being made in larger quantities. New equipment is being ordered, including a development chamber.

R.K.S. in Filler by the use of Stain Technique: R.K.S. was sampled at the blending line and mixed with Marlboro filler at 1%, 3%, 5%, and 12% levels. A two gram sample of filler was used. The average recovery for the various levels of R.K.S. was 84%. This gives a fairly small correction factor.

Ten Marlboro long size cigarettes were randomly selected from our current production and examined for R.K.S. by the stain technique method. The amount of R.K.S. present was much less than was expected. The particle size of R.K.S. in the cigarette was much smaller than the particle size of the R.K.S. from the blending line, which was used in our first evaluation.

The stain technique method will be statistically reevaluated with the small particle size of R.K.S.

71-8101 Comparison of Brands (Wickham, Stewart, Pleasants)

Four comparisons of overseas brands were completed during this period. Each brand was compared with the same brand from the previous month's test. No great difference could be detected.

An eight brand comparison has been undertaken for Operations. The brands will be compared for tar delivery (filtered and unfiltered), nicotine delivery, resistance to draw (both entire cigarette and plug only), and moisture.

Moisture Determinations: Two hundred eighty-six moisture determinations of leaf and filler samples were conducted.

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72-0103 Rapid Method of Analysis (Bennett, Butler)

A study of the smoking machine has shown that the weak point is the valves. These valves were initially designed for use with liquids and do not form a perfect seal for smoking, which requires a tight seal against air leakage. Microscopic examination showed that the "seat" of the valve was irregular, mostly due to grooves, but in some cases due to cavities. Re-cutting of the valves constituted the first attempt to correct this feature. The Horace L. Smith Co. did this work, but the results were not as good as had been anticipated. They then tried to form a "seat" by pressing the valves with a steel ball of the proper size. This appeared to offer some improvement. The testing of these valves led to a more thorough evaluation of both the equipment and procedure used to check them, both before and after incorporating them in the smoking machine. This entire matter is still under investigation.

A check of the puff volume for the eight port constant volume type smoking machine showed an average variance ( $S^2$ ) of only 0.07 c.c./puff for the eight ports. The individual variances ranged from a low of 0.03 to a high of 0.13 c.c./puff. The above puff volumes were measured with all resistances except that of the cigarette and gave a mean value of 35.9 c.c./puff. Another test, with a piece of glass capillary tubing with a 4" resistance to draw inserted to simulate a cigarette, gave a mean value of 35.4 c.c./puff. Seven of the eight ports tested gave lower values with than without the simulated cigarette. The lone exception was the number 1 port, which will be further investigated.

Experiments, either now in progress or destined to be so in the immediate future, have been designed to set up rigid but simple tests to insure effective, accurate smoking of cigarettes by the eight port smoking machine.

74-8101 Services to Marketing Department (Hellams, Bennett, Butler)

Two gross of vials containing bromthymol blue-impregnated strips for demonstrating the no filter feedback advantage of the recess filter were shipped to the Marketing Department. This was done to fill a request that we supply our Milprint Division with the materials and methods necessary to produce some test kits.

In conjunction with our evaluation study of the F.T.C. tentative procedure for "total solids" and nicotine in smoke, a study was begun to determine the moisture content of the "total solids" as collected on filter discs by the F.T.C. procedure. The study will include a moisture determination by the Karl Fischer method and a determination of the amount of material lost by desiccation. Filters will be checked immediately after smoking and after storage under conditions of room temperature and 88% relative humidity.

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Preliminary results showed an average moisture content of 10.7% by the Karl Fischer method and a loss on desiccation of 18.7%. This work will be continued.

Due to an exhaustive study of the eight port smoking machine, the only other experiments carried out in connection with the evaluation of the F.T.C. procedure were aimed at obtaining a simpler extraction method for the effective removal of nicotine from the "total solids" collected on the glass filter discs. A composite chloroform-tar sample was evaporated, and the residue was redissolved in chloroform. Nicotine was added to this solution, and twenty identical aliquots were removed for the nicotine analysis. Ten were extracted by a single partitioning step and the other ten by the F.T.C. procedure, which employs an additional countercurrent extraction. Subsequent analysis by the Griffith method showed the levels to differ by only 0.02 milligram of nicotine and the variances ( $SE$ ) by only 0.004 milligram of nicotine per aliquot. This indicates that while countercurrent extraction is theoretically better than single partitioning, it is not required for this separation.

/mar

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